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 NEWS 4   APR 07 STN is raising the limits on saved answers
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                   information
 NEWS 6   APR 26 USPATFULL and USPAT2 enhanced with patent
                   assignment/reassignment information
 NEWS 7   APR 28 CAS patent authority coverage expanded
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 NEWS 9   APR 28 Limits doubled for structure searching in CAS
                   REGISTRY
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 NEWS 12   MAY 11 BEILSTEIN substance information now available on
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 NEWS 13   MAY 14 DGENE, PCTGEN and USGENE enhanced with increased
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                   introduction of free HIT display format
 NEWS 14   MAY 15 INPADOCDB and INPAFAMDB enhanced with Chinese legal
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 NEWS 15   MAY 28 CAS databases on STN enhanced with NANO super role in
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 NEWS 16   JUN 01 CAS REGISTRY Source of Registration (SR) searching
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 NEWS 17   JUN 26 NUTRACEUT and PHARMAML no longer updated
 NEWS 18   JUN 29 IMSCOPROFILE now reloaded monthly
 NEWS 19   JUN 29 EPFULL adds Simultaneous Left and Right Truncation
                   (SLART) to AB, MCLM, and TI fields
 NEWS 20   JUL 09 PATDPAFULL adds Simultaneous Left and Right
                   Truncation (SLART) to AB, CLM, MCLM, and TI fields
 NEWS 21   JUL 14 USGENE enhances coverage of patent sequence location
                   (PSL) data
 NEWS 22   JUL 14 CA/CAplus to be enhanced with new citing references
                   features
 NEWS 23   JUL 16 GBFULL adds patent backfile data to 1855

 NEWS EXPRESS MAY 26 09 CURRENT WINDOWS VERSION IS V8.4,
              AND CURRENT DISCOVER FILE IS DATED 06 APRIL 2009.

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                                                     ENTRY     SESSION
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chain nodes :
6   7
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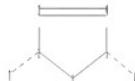
ring nodes :
1 2   3   4   5
chain bonds :
2-7 5-6
ring bonds :
1-2 1-5 2-3 3-4 4-5
exact/norm bonds :
2-7 5-6
exact bonds :
1-2 1-5 2-3 3-4 4-5
isolated ring systems :
containing 1 :

Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:CLASS 7:CLASS

L1   STRUCTURE UPLOADED

=> s l1
SAMPLE SEARCH INITIATED 11:12:52 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED -   8532 TO ITERATE

 23.4% PROCESSED   2000 ITERATIONS   50 ANSWERS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
                        BATCH   **COMPLETE**
PROJECTED ITERATIONS: 165103 TO 176177
PROJECTED ANSWERS: 45344 TO   51238

L2   50 SEA SSS SAM L1

=> s l1 full
FULL SEARCH INITIATED 11:13:01 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED -   172473 TO ITERATE

100.0% PROCESSED   172473 ITERATIONS   45136 ANSWERS
SEARCH TIME: 00.00.01

L3   45136 SEA SSS FUL L1

=> file caplus
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                                                 ENTRY   SESSION
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FILE COVERS 1907 - 20 Jul 2009 VOL 151 ISS 4
FILE LAST UPDATED: 19 Jul 2009 (20090719/ED)
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Jun 2009
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Jun 2009

CAplus now includes complete International Patent Classification (IPC)
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of databases will soon be updated to include new citing references
information. This enhancement may impact record import into
database management software. For additional information, refer
to NEWS 22.

```
=> s l3 and pyrrolidine
         83153 L3
         33258 PYRROLIDINE
          3141 PYRROLIDINES
         34301 PYRROLIDINE
               (PYRROLIDINE OR PYRROLIDINES)
L4         294 L3 AND PYRROLIDINE

=> s l4 and (oxo(L)pyrrolidine)
        172955 OXO
            31 OXOS
        172959 OXO
               (OXO OR OXOS)
         33258 PYRROLIDINE
          3141 PYRROLIDINES
         34301 PYRROLIDINE
               (PYRROLIDINE OR PYRROLIDINES)
          1054 OXO(L)PYRROLIDINE
L5          17 L4 AND (OXO(L)PYRROLIDINE)

=> s l5 and catalyst
        849490 CATALYST
        846073 CATALYSTS
       1089292 CATALYST
               (CATALYST OR CATALYSTS)
L6           2 L5 AND CATALYST
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10572579   mm/dd/yyyy>

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L6   ANSWER 1 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 2005:283464 CAPLUS
DOCUMENT NUMBER: 142:376241
TITLE: Process for preparing N-substituted 2-oxo-
     pyrrolidines such as levetiracetam from
     dihydrofurans and amines
INVENTOR(S):   Surteez, John; Lurquin, Francoise; Diouf, Ousmane
PATENT ASSIGNEE(S):   Ucb, S.A., Belg.
SOURCE: PCT Int. Appl., 19 pp.
     CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC NUM COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005009435	A1	20050203	WO 2004-EP10212	20040913
W:	AA, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,			
	CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,			
	GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,			
	LK, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI,			
	NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY,			
	TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,			
	AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,			
	EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,			
	SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,			
	SN, TD, TG			
AU 2004274157	A1	20050331	AU 2004-274157	20040913
CA 2538933	A1	20050331	CA 2004-2538938	20040913
EP 1667967	A1	20060614	EP 2004-765154	20040913
W:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,			
	IE, SI, LT, RO, CY, TR, BG, CZ, EE, SK			
BR 2004014436	A	20061114	BR 2004-14436	
CN 1852535	A	20061120	CN 2004-80023998	20040913
EA 200600197U	A	20070530	EA 2006-1970	20070228
JP 2007513387	T	20070614	JP 2006-527906	20060307
IN 2006IN01333	A	20070817	IN 2006-001133	20060307
MX 2006007024	A	20061110	MX 2006-3247	20060316
KR 2006121989	A	20061129	KR 2006-705789	20060323
US 2007060523	A1	20070503	US 2004-10310	20070103
PRIORITY APPLN. INFO.:			US 2003-21534	A 20070924
			WO 2004-EP10212	W 20040913

OTHER SOURCE(S):   CAPREACT 142:376241; MARPAT 142:376241
GI

L6   ANSWER 1 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN   (Continued)
     dihydrofurans and amines)
RN   78920-10-2 CAPLUS
CN   2(5H)-Furanone, 5-hydroxy-4-propyl-   (CA INDEX NAME)

REFERENCE COUNT: 5   THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS
     RECORD. ALL CITATIONS AVAILABLE IN THE RE
FORMAT

L6   ANSWER 1 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN   (Continued)

AB   Title compds. [I; R1 = Ra, Rby E3, R4 = H, OH, SH, halo, cyano, CO2H,
     SO3H, Ra, Rb, etc.; X = CONHSR6, CO2R7, cyano; R5,R7 = H, Ra, Rb =
     (substituted) alkyl; Rb = (substituted) aryl, heterocycyl,
     heterocyclalkyl]. were prepared by reaction of dihydrofurans (II, III)

R2,   R21 = alkyl, aralkyl] with R1CR(NH2)X in the presence of H2 and a
     catalyst. Thus, (II)-2-oxobutyramide and
     2,5-dimethoxy-2,5-dihydrofuran were stirred with 1.5 h in aqueos HCl; Na2CO3
was
     added to bring the pH to 9. Pd/C in H2O/EtOH and H2 were added to give
     13% levetiracetam.
I7   532-77-4, 2,5-Dimethoxy-2,5-dihydrofuran 96695-26-0
     RL: RCT (Reactant); RACT (Reactant or reagent)
     (preparation of substituted oxopyrrolidines such as levetiracetam from
     dihydrofurans and amines)
RN   532-77-4 CAPLUS
CN   Furan, 2,5-dihydro-2,5-dimethoxy-   (CA INDEX NAME)

RN   96695-26-0 CAPLUS
CN   3-Furancarboxylic acid, 2,5-dihydro-2-hydroxy-5-oxo-, methyl ester (CA
     INDEX NAME)

I7   78920-10-2P
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
     (Reactant or reagent)
     (preparation of substituted oxopyrrolidines such as levetiracetam from

L6   ANSWER 1 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1956:89309 CAPLUS
DOCUMENT NUMBER: 50:89309
ORIGINAL REFERENCE NO.:
     50:16777d-i,16778a-i,16779a-i,16780a-i,16781a-i,16782a-
     i,16783a-b
TITLE: Ethynylation. V. Reactions of hydrated ethynylation
     products. Dehydration of γ-alkanediols
AUTHOR(S):   Reppe, Walter; et al.
SOURCE: Annalen der Chemie, Justus Liebigs (1955), 596,
     80-158
     CODEN: JLCXA8
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S):   CARREACT 50:89309
AB   XIIa is prepared in quant. yield from 1 kg. (HOCH2CH2)2 stirred and
     heated
     to 165° with 100 g. 80% H3PO4, with fresh IX added and the temperature
     raised slowly to 185° as product distilled, and the distillate dried
     over K2CO3 and redistd., b. 65-7°, d20 0.8886, nD20 1.4065;
     azeotrope containing 5.5% H2O b. 66°. XIIa is also prepared from IX with
     p-MeC6H4SO3H, (CO2H)2, (CH2CO2H)2, maleic anhydride, or
     resorcinol-(HO)3S)2C6H3CHOCH2O evln as catalyst, by heating 35%
     aqueous IX with H3PO4 and NH4Cl in an autoclave to 280° 10 h., or from
     25 g. IX passed over 325 g. precipitated and calcined Cr2O3 at 300-25° or
     CaHPO4 at 300°. Dehydration of the corresponding
     R1R2(OH)CH2CH2C(OH)R3R4 gives the following cyclic ethers (R1, R2, R3,
     R4, and catalyst given): Me, H, H, H (XXXV), H3PO4-H2SO4,
     78-9°; Me, H, Me, H, K2B2O7, 92.5° (azeotrope containing 13%
     H2O, b. 78°), forms with ferrocyanic acid an H2O-insol., colorless,
     crystalline adduct; Me, Me, Me, H3PO4 or K2B2O7, b. 114-17°.
     1,1′-Ethylenediimyclohexanol (120 g.) gives, with 2 mL. 25% H2SO4 in vacuo
     at 150°, 85 g. 2,2/5,5-bis(pentamethylene)-XIIa, m2 93-5°.
     (CH2=CH)2 is prepared by passing 20-5 g. IX and 4-5 g. H2O/h. at
     300-50° over 300 mL. catalyst prepared by mixing 100 g.
     anhydrous NaH2PO4 with 40 mL. H2O, adding 9 g. NaH2PO4 and 20 g.
     graphite,
     evaporating with continuous stirring, heating finally to 160°, and
     breaking to suitable size. The same catalyst, with 98% H3PO4
     instead of Bu phosphate, gave 90% (CH2=CH)2 from XIIa at 280°; 1800
     g. XXXV gives 510 g. MeC8(CH3H2CH2, b. 40°, and 710 g. recovered
     XXXV. (MeCH(OH)CH2)2 (500 g./day), passed at 280-300° over
     catalyst prepared by adding 125 g. K2(OH)3 to 700 mL. H2O and 700 g.
     90% H3PO4, stirring 2-3 h. at 110-15°, adding 185 g. NaH2PO4 and 43
     g. BuNH2, cooling, adding 320 g. 34.4% water glass, ball, milling,
     evaporating
     in vacuo at 280°, and crushing, gives 240 g. (MeCH(=CH)2, b.
     77-9°. 2,5-Dihydrofuran (17 g.), prepared from 50 g. XXV and 2 g.
     20% H2SO4 at 125-70° and 18 mm., b. 63.5° (forms azeotrope
     containing 7.5% H2O). XXV is also dehydrated by (CO2H)2 at 175°, by
     passing in HCl at 160° or by passing it over Al2O3 at
     240-50°. 2,5-di-Me-XIIb, b. 90-3°, and
     2,2/5,5-bis(pentamethylene)dihydrofuran, b16 123-5°, are prepared
     similarly. 1,2,4-Butanetriol (700 g.) and 30 mL. concentrated H2SO4 at
     30 mm.
     and 100-15° treated with an addnl. 1500 g. triol and distilled during
     36 h. gave 1750 g. 3-HO-XIIa (XXXVI), b740 183°, bD20 97-5°;
     acetate, b12 64°. XXXVI (85 g.) distilled at -5° to 160 g.
     (OCl)2, warmed to 20° in 1 h., and swept with dry air, the residual

chlorofumarate [XCVII] treated with 100 mL. NH4OH at 20-45°, then a stream of HCl gas, and worked. with EtO2, and the mut. evapd. gives 100 g. XXXVI urethane [C5H9NO3], m. 57° (from C6H6). Prepd and XXXVII give the NH-ph urethane, m. 112°, and N2H(CH2)6NH2 gives N,N'-hexamethylenebis(tetrahydro-3-furyl urethane), m. 113°. XXXVI [176 g.], 3 g. Na, and 135 g. CH2=CHCH gives β-(tetrahydro-3-furyloxy)propionitrile, bd 108-10°.

HOCH2CH2[CH3OH3]2NMe and concd. H3SO4 at 190° give 1-methyl-4-hydroxy-XIIa, m. 98°, bd3 90-1°.

2,3-Dichloro-XIIa [XXXVIII], bd3 67° [1200 g.] is prepd. by passing Cl2 into 1506 g. XIIa at 0-10° for h.; 500 g.
2-(4-chlorobenzoyl)-3-chloro-XIIa [XXXIX], bd3 145-55°, is also
formed. XXXVII [1300 g.] and XXXIX [294] are also prepd. by passing XIIa
and
Cl dild. with N ever glass beads at 100°; XXXIX is prepd. in quant.
yield by passing HCl into 136 g. XIIa and 280 g. XXXVIII at 20-30°
in ice... (dense fine print continues)

L6   ANSWER 2 OF 2 CAPLUS   COPYRIGHT 2009 ACS on STN   (Continued)
1G2-1"; thus VII gives at varnus to warm milk of lime and the vapors condensed and radiated. gives 24 g. 3,4-oxido-XIIa (XLVII), b. 141°, nH 45°   XLVII, KOH, and Al2O3 at 250° gave monoesters of 2,4-dihydroxy-XIIa (R and oesta: given): H, bd3 163'; Me, bd3 197'; Et, bd3 215'; Bu, bd5
134'; HOCHₓCH₂; bd2 281'; Ph, bd5 231'; PhCH₂, b0.6
1G7' 2,3-dimethyl-3-hydroxy-δ-methoxy-XIIa, bd3 102'.
XLVII (96 g.) rooled and 620 g. 25% NHY added gives
2-hydroxy-4-amino-XIIa, bd1 147', n. 78'.
Amino derivs. prepd. from XLVII and XLXPH at 200° are (R1 and R2 given:: oct=deoyl; A, bd.5 325'; Ph, H, m. 147° (from ligroine); Ph, Me, m. 67-4'; bd.5 175'; pyrrolidine , bt7 153'. XXV bis²˙²trihydro-3-furyl) ether, bd 140-56°, and 72 g. Cl added simultaneously at 15-30° during 2 h. to 2300 mL. H2O give 284 g. crude chlorohydrin which, added to 76 g. powd. Ca(OH)2 in 5QQ mL. H2O, gives 150 g. 1,7-oxido-1,4-butylene bis(tetrahydro-3-furyl) ether, bl1 112-53'. XXV (163 g.), treated at 5-10° with 247 g. HCl gives 542 g. 5-chloro-XIIa, b17 70-5'. AlII (180 g.) and 9 g. powd. "Na (dimethylnaphthalenesulfonate" in 10 l. H2O treated at room temp. with 265 g. Cl disld. with air gives an unidentified tetrachlorobutane, bd9 45-5', and 287 g. tetra(2-CH3ClCHClCO(OH)CHCl(CH2)CLv)Cl(,, 2 isomers, bl.2 74-6' and bl
1Q3-4'. 12VIIa (161 g.) and 60 g. powd. Ca(OH)2 in 70 mL. H2O shaken 15 min. gives 72 g. of a mixt., bd 18-71°, of 1,4-dichloro-2,7-oxidobutane and 1,2-dichloro-1,4-oxobutane. H2O2 (110 q. 40%) added to 115 g. AlII, 200 g. AsO6, and 0.04 g. OsO4 gives, after 2Q h., 56 q. CH2ClCH(OH)(CHCl)O, m. 117'; thus (80 q.) in 1 l. XIIa refluxed 3 h. with 60 q. NaOH gives 30 g. 1,2,3,4-dioxidobutane, bd3 50-1'. XXV (3QQ q., 97%) in 1 l. AcOH chlorinated 5 h. at 0-10°, gives 280 g. (CHClₓCHOCl), bl 134-6', m. 70'. (CH₂CH₂OH)₂, passed similarly in CH2Cl2, m. 87' (from C6H6), bl.3-1.5 140-52'. Et1 passed into 400 g. HOCH(CH₂)O(CH₂CH₂OH and 8 g. AcOH at 90-100° until 300 q. has been absorbed gives 420 g. CH₂Cl(CH(OH)CH(OH)CHCl, bl3 95-130°; 400 g. of this in XIIa and 112 g. NaOH in 250 mL. H2O at 15-20° give 240 g. 1-chloro-3,4-oxidobutane, bd3 63', 190 g. of which heated to 120-70' with 90 q. powd. NaOH gives 50 g. butadiene monoxide, b. 68'. XXV (80 q.) and 89 q. anthracene in 3 h. at 250° under N gives 9,10-endo-(2',3'-butene-1',4'-diol)anthracene (XLVIII), m. 221' (from air.); diacetate ester, m. 151' (from air.-H2O). Similarly XXIVa gives, with anthracene, 9,10-endo-(3',4'-tetrahydrofuryl)anthracene, m. 180' (also obtained from XLVII) and 78 H2O4 refluxed 7 h., and, with 9,10-dichloro anthracene, 9,10-dichloro-9,10-endo-(3',4'-tetrahydrofuryl)anthracene, m. 145'. [MeC(OH)CH₂]2 passed 90-95% pyrrolidine XIIa gives
9,10-endo-(3',4'-hexane-2',5'-diol)anthracene, m. 213'. XIIa (10 q./b./l. catalyst) and NH3 (molar ratio 1:20, resp.) passed over Al2O3 at 700-20° gives 90-5% pyrrolidine (XLIX), b. 87-8' (N-Ac deriv., b. 112-14', p-O2NC6H4CO deriv., m. 92-3'), 1,-3-(butyryl-XLIX, b. 152-4', bl4 64-5' (picrate, m. 107' (from alc.)), 1-(4-aminobutyl)-XLIX, b. 115-19' (picrate, m. 167' 67-8; deriv., bl 180'), 1,4-di(1-pyrrolidinyl)butane, bl 100' (dipicrate, m. 158'), dihydrate, m. 72'); N(CH2CH2CH₂CH3), bl 120'. HO(CH2)4OH

L6   ANSWER 2 OF 2 CAPLUS   COPYRIGHT 2009 ACS on STN   (Continued)
similarly gives 858 XLIX and 6-8% high-boiling compds. contg. NH(CH₂CH₂CH₂)₂. b. 155-60° (picrate, m. 246°). XLIX or CH2Cl(CH2)4OH and KNH2 over Al2O3 or Th02-Al2O3 at 700-20° give the following 1-derivs. of XLIX (substituent given): Me, b. 79-81°, Et, b. 105-7', Pr, b. 129-31', bd5 32-5', Bu, b. 154-6', bd5 48-50'; n-C10H21, bl 14-16'; (CH2OCH3), bd2 55-7'; 1,2-di(1-pyrrolidinyl)ethane (formed with the preceding compd.), b0 90-100' (hydrate, m. 52'); Ph (L), bl 192-4', m. 11' (L couples with disco compds.); with NH22 L gives a green g-OH deriv., m. 128'; γ-naphthyl, bd 4 177'; cyclohexyl, b. 217-19'; 2-pyridyl, bl1 100-1'; 3-ethyl-6-methyl-2-pyridyl, h2O 130-70'; 8-quinolyl, b0.8 158-60'. Analogously 222 g. HOCH2CH2OCH2CH2OH and 245 g. PhNH2 give 70 q. 1-phenylpyrroline. [MeCH(OH)CH2] gives 1-phenyl-2,5-dimethyl XLIX, b0.6 80-6'; tetrahydro-pyran with NH3 gives piperidine and 1-(pentanyl)piperidine, b13 81-8'; PhNH2 gives 1-phenylpiperidine, bl1 175-5'. L (17 q.) added at 10° to 50 q. PhNHₓCHO and 69 q. PoCl3, the mixt. warmed several hrs. to 40-5', and decompd. with ice gives 4-(1-pyrrolidinyl)benzaldehyde, m. 81° (from EtO2-petr. ether); semicarbazone, m. above 700°. Similarly L and CHCl2 in CHCl3 at 5° give 4-(1-pyrrolidinyl)benzoyl chloride, sublming at 100-50°/0.1 mm.; hydrolyzn gives the acid, m. 194-5° (from aln.). HOCH(CH OH)CH₂CH₂OH (160 q.), 115 g. PhNH2, and 500 g. PhNH2.HCl heated

L2
 h. at 190° gives 50 q. 3-hydroxy-L, bl 139-4',
1-m-tolyl-2-hydroxy-XLIX prepd. similarly from m-MeC6H4NH2, b0.7-2.5
111-40'; 2-methyl-L, from 172 q. XQGV, 170 q. PhNH2, and 70 q. fullers earth 11 h. at 250°, bl3 125-35'. XLIX (93 q., 90%), 60 g. m-MeC6H4OH, 60 mL. concd. HCl, and 200 mL. H2O 12 h. at 160° give 50 q. 1-(m-hydroxy)-L, m. 135'. 1-β-naphthyl-XLIX, m. 92', 150 q. from (16 q. β-naphthol, 200 q. XLIX, and 20 q. fullers earth 6 h. at 200°), 1,6-di(1-pyrrolidinyl)naphthalene, m. 240', was prepd. similarly, in 90 q. yield from 70 q. 1,4-dihydroxynaphthalene and 70 q. XLIX. Ethylene oaiie (246 q.) passed at below 50° into 755 q. XLIX and warmed 1 h. at 50° gives 213 q. 1-(2-hydroxyethyl)-XLIX, bl 57-62', 100 q. 1-(2-(2-hydroxyethoxy)ethyl)-XLIX, bl 91-4°, and 29 q. 2-(2-hydroxyethoxy)ethyl β-(1-pyrrolidinyl)ethyl ether, bl 120-4'. VIII (100 q. 30%), and 142 q. XLIX in 1 h. at 52° give 130 q. di(1-pyrrolidinyl) methane, b20 81-5' (1-Me-XLIX gives bis(2-methyl-1-pyrrolidinyl) methane, bl 57-67'). The following 1-substituted XLIX are prepd. indetthinaet given): CH2CH2OCON2, b0.5 66-8°, CH2CH2OH, b20 105-8' [this with 100 atm. H2 at 100° gives the same ammine, C7H18N], b10 36' isocaproiric, m. 145', mono p-aminobenzoyl deriv., m. 159'); CHO, bl0 112-14', and a compd. gives, 2 mol XLIX and 1 mol CO (from XLIX and CO); CONH2, m. 213' (from alc.). CS2 (144 q.) and 117 q. XLIX reacted with MeCHO give 1008 N-pyrrolidinyldithiocarbonoic acid Me ester, b9 155-8°, m. 90°. p-Toluenesulfonylpiperate (137 q.), and 71 g. XLIX, react on mixing to give Me-(1-pyrrolidinyl)methyl-p-toluenesulfate, Cl(NH4NO3 m. 214' (from alc.). XLIX (284 q.), and 142 q. (CH2ClCH2)2O heated 12 h. at 160° give 170 q. bis[2-(1-pyrrolidinyl)ethyl] ether, bl2 166-71'. The following

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compds. are prepd. by conventional methods from XLIX and halo (usually Cl):
   compds. (R = 1-pyrrolidinyl): (CH2ClCH2)2, b22 146-52';
   [HOCH2CH2CH2]2, bl 190-200°, m. 61', p-O2NC6H8 (LI), m.
   164° [hydrogenation over Ni-Cr2O3 at 70-80°, 200 atm. p-H2N
   analog (LII), bl 140', m. 35°]; p-O2NC6H4CH23, hydrogenated
   to the p-NH2 analog, bd 129', m. 51' (from ligroine);
   1-(3-quinolyl)pyrrolidine, m. 96-8' (from ligroine);
   n-O2NC6H4SO2R, p-AcNHC6H4SO2R, m. 177' (from H2O); p-H2NC6H4SO2R,
   m. 168' (from H2O) (by hydrolysis of the preceding compd. with 5N
   N2SO4); 3,4-Cl2C6H3SO2R, m. 113' (from ligroine);
   3,5,2-Cl2(HO)C6H3SO2R, m. 146' (from 70% alo.);
   1,1,4,6-Me2HO(C6H)SO2R)2, m. 239' (from ligroine);
   3,4-Cl2C6H3SO2R, m. 126'. L21 sulfate (80 q.) in 250 q. concd.
   H2SO4 and 115 mL. HNO treated at 110° with 72 q. L2, and 95 q.
   glycerin, H2O stdtd. to raise the temp. to 149' and held there 6-7
   h. gives 1-(4-cyclnopil)-XLIX, bd 145-75', m. 117' (from
   C6H6-ligroine). The sulfo(biroid (20 q.) from HOSHCl and 4,1-ClMeC6H3OH
   refluxed 2 h. with 40 q. XLIX give 1,5,4-ClMe (HO)C6H2SO2R
   (R-1-pyrrolidinyl), m. 126' (from ligroine).
   2-Mercaptobenzothiazole (334 q.) in 1500 mL. of H2O treated with 426 q.
   XLIX, then 5 l. 15% NaOCl given 1-benzothiazolylsulfenepyrrolidide,
   Cl1H12N2OS2 (L3II), m. 170'. VIII (400 q., 30%) and 280 q.
   iso-PrCHO added with stirring and cooling to 284 q. XLIX and stirred
   several hrs. at 40-50° give
   2,2-dimethyl-3-(1-pyrrolidinyl)propionaldehyde, b14 85°, this with
   NH3 and 150 atm. H at 150° over Ni give
   2,2-dimethyl-3-(1-pyrrolidinyl)propylamine, b0.5 55-7'. XLIX (200
   q.) and 200 atm. H 12 h. at 200° over 40 q. Raney Co give 27 q.
   1-Bu-XLIX, b. 156-4' (picrate, m. 125-7'), and a small amt.
   of (CH2CH2R)2 (R = 1-pyrrolidinyl), b5 134', similar reaction in
   the presence of H2O2 gives 50 q. 2-Bu-D1ILX, b. 154-6'
   (picrate, m. 122-3'); at 200° over Raney Ni HCHO and XLIX and
   1-H2N(CH2)4-XLIX, b10 88-119' (picrate, m. 125'), and
   1,4-di(1-pyrrolidinyl)butane, b00 121-4' (picrate, m. 157'),
   are formed. IX (190 q. 50% aq. soln.), astd. with NH3 and passed during
3
   h. over 250 mL. 5% ThO2 on Al2O3 over which 200 l./h. NH3 is circulated
   gives 80 q. unchanged IX and 30 q. pyrrole (LIV), b. 130'. A mixt.
   giving. 2/3 1-Me-LIV, b. 112-13', and 1/3 2-Me-LIV, b.
   147-8', is prepd. in 63% yield from 1-Me-XLIX passed at 500°
   with N over a ZnO-CuO-Al2O3 catalyst. 2-Me-LIV is also prepd.
   in 83% yield from 1-Me-LIV over Ag shps at red heat, L dehydrogenated
   similarly gives 808 1-Ph-LIV, m. 51-8'; rearranged as above to
   2-Ph-LIV, m. 122-3'. XQGVa (180 q.), for an argon. XXV or
   HOCHₓCH₂CH₂(CH2OH) and 228 q. PhNH2 over ThO2-Al2O3 at 300° gave
   1-phenyl-2,5-dihydropyrrole, bd 85-97', m. 88' (from MeOH);
   this, 50 q. in 200 mL. C6H6 added to 50 q. PhNHₓCHO and 115 q. PoCl3 in
   100 mL. C6H6, held 2-3 h. at 0-20° and 12 h. at room temp., givnz
   4-(1,5-dihydro-1-pyrrolyl)benzaldehyde, m. 90-3' (from
   cyclohexane); semicarbazone, m. 270° (from alc.). XIIa (200 q.,
   94%) added to 642 q. 65% HNO3 and 2 q. NaNO2 at 25' gives 128 q.
   (CH2ClCH2)2 (ia anhydride (LIVa) is prepd. by passing this over Al2O3 or
   SiO2 at 275° and 120-60 mm. HOCH(CHₓ)₃CH₂OH is prepd. similarly
   from 2-Me-XIIa; the anhydride, m. 30-5', bl 105'. XLXVA
   (100 q.), passed at the rate of 6 q. and 20 l. air/h. over 100 mL.
   catalyst (prepd. by heating 110 q. (NH4)2MoO4, 72 q. vanadic acid,
   and 60 q. ThO2 1 h. with 1 l. 44 (CO2N)2, adding 20 q. H84Cl), then 500 q.

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pumice, evapd., and heating 2 h. at 300°) gives 100-70 q. maleic acid (LV) and anhydride; 100 q. XIIa similarly gives 90-90 q. LV and anhydride. Cl (70-80 q.) passed into 100 q. LIVa at 150-80° gives 60 q. LV anhydride and 40 q. unchanged LIVa. Monochloro-LV anhydride is prepd. from maleic anhydride (or LIVa, Cl, and FeCl3 at 160-80°; 100 q. LIVa, same FeCl3, and 240 q. Cl at 160-80° give 152 q. dichloromaleic anhydride. LIVa (100 q. 90%), reacted at 190-200° with 3 q. NaOEt gives 260 q. y-oxoglutaric acid disiloxane, bd 170°; 300 q. of this refluxed 60 h. with 600 mL. EtOH and 70 q. concd. H2SO4 gives 300 q. diester of y-oxo-pimelic acid, bl1 165°. Hydrogenation of the diluatone in alk. soln. at 160°, 200 atm. gives 90% α-hydrocopimelic acid; 500 q. of the dilactone, and 350 q. MeNO3 in 1500 mL. H2O hydrogenated over 35 q. Raney Ni and 35 q. Cu chromite at 280°, 200 atm. give 220 q. pimelic acid.

IT   96-02-6P, Maleic anhydride, chloro- 108-31-6P, Maleic anhydride 1112-17-4P, Maleic anhydride, dichloro-
     RL: PREP (Preparation)
     (preparation of)
CN   96-02-6 CAPLUS
CN   2,5-Furandione, 3-chloro- (CA INDEX NAME)

[structure]

RN   108-31-6 CAPLUS
CN   2,5-Furandione (CA INDEX NAME)

[structure]

RN   1112-17-4 CAPLUS
CN   2,5-Furandione, 3,4-dichloro- (CA INDEX NAME)

[structure]
     Cl   Cl

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